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ON THE REACTIONS OF THE FORMAMIDINES.

Syntheses of Isoxazolon, Isoxazol, Cyanacetic and Benzoylacetic Acid Derivatives.

By F. B. DAINS and E. L. GRIFFIN.

IN previous papers it has been shown that compounds containing a methylene group, CH_2, such as acetoacetic ester, benzoylacetic ester and cyanacetic ester, are capable of reacting with substituted formamidines yielding derivatives of the type XYC : CHNHR, in which the two methylene hydrogens are replaced by the grouping :CHNHR. Phenyl isoxazolon, another compound containing methylene hydrogen, reacts in the same way, yielding substituted aminomethylene derivatives of phenylisoxazolon. The compounds made by this reaction were: 3-phenyl-4-anilidomethylene-5-isoxazolon, 3-phenyl-4-o-toluidomethylene-5-isoxazolon, 3-phenyl-4-p-toluidomethylene-5-isoxazolon, 3-phenyl-4-m-toluidomethylene-5-isoxazolon, 3-phenyl-4-o-anisidomethylene-5-isoxazolon, 3-phenyl-4-p-anisidomethylene-5-isoxazolon, 3-phenyl-4-p-phenetidomethylene-5-isoxazolon, 3-phenyl-4-pseudocumidomethylene-5-isoxazolon, 3-phenyl-4-nitroanilidomethylene-5-isoxazolon, and 3 phenyl-4-p-bromanilidomethylene-5-isoxazolon.

It was found that derivatives analogous to these could be formed by the reaction of either benzal-or anisal-methylisoxazolon on the formamidines, the substituted aninomethylene derivatives of methylisoxazolon and benzal-or anisal-amines being obtained. From benzalmethylisoxazolon were obtained 3-methyl-4-anilidomethylene-5-isoxazolon, 3 methyl-4-o-toluidomethylene-5-isoxazolon, 3-methyl-4-p-toluidomethylene-5-isoxazolon, 3-methyl-4-m-toluidomethylene-5-isoxazolon, 3-methyl-4-p-anisidomethylene-5-isoxazolon, 3-methyl-4-o-anisidomethylene-5-isoxazonon, 3-methyl-4-p-phenetidomethylene-5-isoxazolon, and 3-methyl-4-m-xylidomethylene-5-isoxazolon. From anisal methylisoxazolon and formamidines were made 3-methyl-4-anilidomethylene-5-isoxazolon, 3-methyl-4-p-anisidomethylene-5-isoxazolon, and 3-methyl-4-pseudocumidomethylene-5-isoxazolon. Anilidomethylememethylisoxazolon and anilidomethylene phenyl-isoxazolon were found to react with bromine, with the loss of hydrobromic acid, forming monobrom derivatives, which on solution in alcohol go over to p-bromanilido-methylene-methyl-isoxazolon and p-bromoanilido methylene-phenyl-isoxazolon. With dilute alkali these isoxazolon derivatives dissolved and formed a salt from which the same isoxazolon could be regained by adding hydrochloric acid. With heat and standing, however, this breaks down, giving the amine and another salt.

When hydroxylamine acts on the amides of aminomethylene-aceto or benzoylacetic acids, there are formed 5-methyl or phenyl-isoxazol 4-carboxylic acids. These isoxazols, under the influence of alkalies, rearrange to form substituted amides of acetyl or benzoylcyanacetic acid. To study these reactions several compounds were made, including the following: the o-totylamid of 5-phenylisoxazol-4-carbocylic acid, and from this by the action of NaOH benzoyl-cyanacet-o-toluid; p-tolylamide of 5-phenylisoxazol-4-carboxylic acid, and from this benzoyl-cyanacet-p-toluid; the p-anisylamid of 5-phenylisoxazol-4-carboxylic acid which with the alkali gives benzoylcyanacet-p-anisid; the anilid of 5-phenylisoxazol-4-carboxylic acid, which likewise rearranges; also, the O-totyl and p-totyl amides of 5-methylisoxazol-4-carboxylic acid were made, giving in the same way acetcyanaceto-o-toluid and p-toluid. The anilid of 5-methylisoxazol-4-carboxylic acid was separated with difficulty, but reacted in the same way to form acetcyanacetanilid.